Formulation of a fat surfactant vehicle containing a fragrance.

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Inventor:

MCDERMOTT KEITH J (US); GUENIN ERIC P (US); TEFFENHART JOHN M (US); SHEFER SHMUEL D

(US); TAN CHEE-TECK (US); SMITH LESLIE C (US)

Applicant:

INT FLAVORS & FRAGRANCES INC (US)

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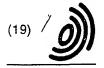
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Abstract of EP0679715

A method is disclosed for producing a fragrance containing solid particles for incorporation into laundry detergent by selecting a fat component such as a fatty acid glyceride, heating said fat component to an elevated temperature sufficient to form a molten melt thereof, selecting a solid surface active agent from the group consisting of SPAN TM surfactants with an HLB of 4.3 to 8.6, heating the surface active agent to form a molten melt thereof and then combining the melts with an aroma chemical to form a mixture. The melt is rapidly cooled to form a solid material.

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(54) Formulation of a fat surfactant vehicle containing a fragrance

Formulierung einer Zubereitung aus Fett, Tensid und Parfüm Formulation d'un agent gras/détergent contenant un parfum

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(73) Proprietor: INTERNATIONAL FLAVORS & FRAGRANCES, INC.
New York New York 10019 (US)

(72) Inventors:

McDermott, Keith J.
 Bridgewater, New Jersey 08805 (US)

Guenin, Eric P.
 Piscataway, New Jersey 08854 (US)

Teffenhart, John M.
 Edison, New Jersey 08817 (US)

Shefer, Shmuel D.
 East Brunswick, New Jersey 08816 (US)

 Tan, Chee-Teck Middletown, New Jersey 07748 (US)

 Smith, Leslie C Jamesburg, New Jersey 08831 (US)

(74) Representative: Brown, John David et al FORRESTER & BOEHMERT Franz-Joseph-Strasse 38 80801 München (DE)

(56) References cited:

EP-A- 0 036 720 US-A- 4 209 417 DE-A- 4 216 453

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Description

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[0001] The present invention relates to a formulation of a fat and a solid surface active agent for use as a carrier for an aroma chemical or fragrance for the purpose of imparting a fragrance to a laundry detergent composition containing the fat/surface active agent formulation used to increase substantivity of fragrances on fabrics. In another aspect, the present invention relates to a method of formulating a fat and surface active agent carrier for one or more aroma chemicals.

[0002] The method of the present invention enables the production of fragrances containing solid particles of improved substantivity for use in a variety of laundry detergents.

[0003] US-A-4209417 discloses perfumed particles comprising water-insoluble perfume, water-soluble polymer, and emulsifier. The particles are comprised of a continuous polymeric matrix having dispersed substantially uniformly therethrough perfume/emulsifier droplets.

[0004] EP-A-0036720 discloses particles containing perfume and a matrix material, the matrix material being a cationic component, optionally together with a nonionic component.

[0005] It is an object of the present invention to provide improved substantivity of fragrances by utilizing a suitable carrier to bring the fragrance to the laundered clothes.

[0006] It is a further object of the present invention to provide improved powdered laundry formulations which result in improved substantivity of fragrances.

[0007] In obtaining the above and other objects, one feature of the present invention resides in formulating a fat and solid surface active agent carrier for a fragrance to be used in a laundry detergent.

[0008] According to the present invention there is provided a method for producing an aroma chemical-containing solid particle of improved substantivity for incorporation into laundry detergents characterized in that the process steps, in sequence, consist essentially of:

- (i) selecting a fat component from the group consisting of glycerol which is mono, di or tri substitued with a saturated or unsaturated organic moiety of 3 to 30 carbon atoms;
- (ii) heating said fat component to an elevated temperature sufficient to form a first molten melt thereof;
- (iii) selecting a solid surface active agent from the group consisting of a SPAN® surfactant of HLB 4.3 to 8.6, said SPAN® surfactant being defined as a mixture of compounds having the structure:

wherein R is C₁₁-C₁₇ alkyl or alkenyl;

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- (iv) heating said surface active agent to form a second molten melt thereof;
- (v) combining said first and second melts with one or more aroma chemicals and uniformly dispersing said one or more aroma chemicals in the combined melt of said fat component and said surfactant;
 - (vi) rapidly cooling said melt to form a solid material containing said fat component, said nonionic SPAN® surfactant and said one or more aroma chemicals; and
- (vii) forming solid particles thereof to obtain an aroma chemical-containing particulate aroma chemical carrier,

wherein the fat component is present in an amount of 40 to 99% by weight, and the surfactant is present in an amount of 1 to 60% by weight, excluding the quantity of aroma chemical, and wherein the aroma chemical is present in an amount of 1 to 40% by weight based on 100 parts by weight of combined fat component and surfactant.

[0009] Thus the method for producing a fragrance containing solid particles of improved substantivity for incorporation into laundry detergents may be carried out using the sequential process steps of:

- (i) selecting a fat component from the group consisting of glycerol which is mono, di or tri substituted with a saturated or unsaturated organic moiety of 3 to 30 carbon atoms;
- (ii) heating the fat component to an elevated temperature sufficient to form a first molten melt thereof;
- (iii) selecting a solid surface active agent from the group consisting of a SPAN® surfactant of HLB 4.3 to 8.6;
- (iv) heating the surface active agent to form a second molten melt thereof;
 - (v) combining the first and second melts with one or more aroma chemical and uniformly dispersing the aroma chemical in the combined melt of the fat component and the surfactant;
- (vi) rapidly cooling the melt to form a solid material containing the fat component, the nonionic SPAN® surfactant and aroma chemical; and
 - (vii) forming solid particles thereof to obtain a fragrance containing the aroma chemical carrier.
- 45 [0010] The SPAN® surfactants are defined as mixtures of compounds having the structure:

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wherein R stands for fatty acid residues.

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[0011] The fat component is present in the amount of 40 to 99% by weight, and the surfactant is present in the amount of 1 to 60% by weight, excluding the quantity of aroma chemical. The aroma chemical is present in an amount of 1 to 40% by weight based on 100 parts by weight of combined fat component and surfactant.

[0012] It has been found useful for the fat component to contain 10 to 20 carbon atoms.

[0013] Preferably, the fat component is selected from natural fats obtained from solid waxy oils from soybean, palm, corn, cottonseed, safflower and coconut plant sources.

[0014] Typically, the fat has the formula:

wherein R can be the same or different organic group containing 5 to 30 carbon atoms.

[0015] It is further disclosed to provide carrier matrices based on fat and surfactants for the fragrance which is carried to the laundered clothing.

DETAILED DESCRIPTION OF THE INVENTION

[0016] It has been found that improved substantivity of fragrances can be achieved by formulating a fat and solid surface active agent which is then introduced into a laundry detergent. By a careful selection of the fat and solid surface active agent, it is possible to achieve improved substantivity of the fragrance.

[0017] In carrying out the invention, the formulation of the fat and solid surface active agent is begun by selecting a fat component in the amount of 40 to 99% and combining it with a low HLB surfactant such as a fatty acid ester in the amount of 1 to 60% surfactants suitable for the purposes of the invention are those sold under the trade mark SPAN® which are non-ionic surface active agents described in more detail below. It has been determined that the HLB value ranges from 4.3 to 8.6 in order to obtain the results described herein. These two components are melted by heating each to a sufficiently elevated temperature to form a melt and mixed at a temperature above the melting point of the

highest melting component. To the resulting melt, there is then added one or more aroma chemicals to impart the desired fragrance in the amount of about 1 to 40% by weight based on 100 parts by weight of the molten mix of fat component and surfactant. After thorough mixing to ensure uniform dispersion of the aroma chemical in the fat/surfactant mixture to form a homogeneous mixture, the mixed components are subjected to fast cooling to yield a solid material which is the fat/surfactant fragrance carrier. This solid carrier is then ground to a suitable particle size and then mixed with a conventional laundry detergent base formulation to produce the final detergent formulation to be used by the customer.

[0018] One suitable method for making the fragrance carrier material is to premelt the fat component by heating at about 100°C and the surfactant at about 70°C. Generally, the melt is formed by heating the components above the melting temperature of the highest melting component. Any suitable heating equipment can be used for this purpose such as a scraped wall steam jacketed vessel. The two melts are then mixed together to ensure an adequate and uniform dispersion of both materials in each other. Suitable mixers known in the art can be used for this purpose. The aroma chemical or fragrance is then added and then mixed with the molten mixture. The molten mixture is then rapidly cooled on a suitable cooling apparatus such as chilled drums or cooled belt to produce a solid material. The product is then ground through a 2 mm screen in a conventional grinding apparatus sich as a Cumberland mill with cryogenic cooling in order to remove the heat generated by grinding. The result is particles of about 1 mm in diameter.

[0019] Alternative methods for forming the final product particles can be used such as spray chilling, drum chilling and extrusion.

[0020] Examples of materials that can be used in accordance with the invention as the fat component are the following fatty materials:

triglycerides which are esters of saturated and unsaturated fatty acids.

[0021] Illustrative of these compounds are those represented by the formula.

wherein R can be the same or different aliphatic organic groups containing 5 to 30 carbon atoms (representing stearic acid esters, palmitic acid esters, oleic acid esters, linoleic acid esters and the like).

[0022] Surfactants suitable for purposes of the invention are those sold under the trademark SPANS which are nonionic surface active agents represented by the formula:

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wherein R represents the fatty acid residues of the SPAN® 20, SPAN® 40 and SPAN® 80. SPAN® 20 contains the lauric acid residue. SPANS 40 contains the palmitic acid residue. SPAN® 80 contains the oleic acid residue.

[0023] The SPAN® type materials are partial esters of the common fatty acids (lauric, palmitic, stearic and oleic acid hexitol anhydrides (hexitans and hexides) derived from sorbitol.

[0024] Specific examples are sorbitan monolaurate, monooleate, monopalmitate, monostearate. Diesters and triesters of sorbitan are also suitable.

[0025] Materials used in the examples were:

DURKEY® D17 partial hydrogenated soybean oil as the fat.

[0026] Certain compositions of fat/surfactant were added to powdered laundry detergent and improvements in substantivity of fragrance on wet laundry were observed. All tests were conducted with panels of 18 to 20 people. Control samples were prepared by adding 0.3% fragrance to the laundry detergent base and tumbling in a Turbula mixer until the fragrance was fully dispersed. Carrier samples were prepared by blending the appropriate amount of carrier with laundry detergent to yield 0.3% fragrance then tumbling in the Turbula mixer. Washes were made on a laundrometer at 37°C with one wash and one rinse cycle.

[0027] Samples of "reference" and "sample" were presented to each panelist and they were asked to compare the fragrance intensity of the reference to the sample and indicate if the sample is:

- 1 much stronger;
- 2 stronger;
- 3 slightly stronger;
- 4 same;

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- 5 slightly weaker;
- 6 weaker; or
- 7 much weaker;
- 45 than the reference.

[0028] Results were tabulated by dividing the number of stronger responses by the total of responses to yield "% stronger".

[0029] In the panel tests, significant increases in substantitivity on white polyester/cotton (70/30) cloth were obtained using carriers formed of Fat/SPAN® 40/Fragrance in the proportion of 60/20/20% by weight. Good results were also observed with formulations wherein these proportions were changed to 40/40/20% by weight.

[0030] The fragrance components or aroma chemicals were combinations of proprietary materials identified as "a citrus type" laundry fragrance" and a "floral type" laundry fragrance.

[0031] Laundry bases used in these tests were:

- 1 powdered detergent with phosphate, inorganic bleach and TAED bleach activator;
- 2 powdered detergent containing phosphate; and

3 - powdered detergent containing nonionic surfactant and zeolite.

[0032] Although not wishing to be bound by any theory, applicants believe that when washing, the fat protects the fragrance from being removed from the wash water by the detergent. The surfactant provides a slow emulsification of the fat/fragrance which allows interaction and deposition of the fragrance on the clothes. This deposition is possible due to the Van den Waals bonding between the fat and the clothes.

[0033] 14 Week stability tests were also carried out on these samples and were found to be stable in the above bases and in some cases with improved fragrance stability.

[0034] The panel testing procedures are described in the following examples.

EXAMPLE

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[0035] The samples prepared were tested for effectiveness. A group panel test was organized consisting of 5 people. [0036] The laundry samples were prepared at a 0.3% effective fragrance concentration using a citrus type laundry fragrance.

[0037] Fabric swatches were 65/35 polyester/cotton.

[0038] Four pieces of 7.6 cm x 7.6 cm (3" x 3") fabric swatches were placed in 1 liter launderometer canisters with steel ball bearings.

[0039] 750 ml of water was added. The detergent and carrier were added to each canister and sealed. In addition, an oil-on-detergent reference was also used.

[0040] The group test panel results revealed that the samples containing the fat/surfactant (SPAN® 40) performed better than the others.

[0041] Samples evaluated in this example were as follows:

- Oil absorbed on detergent;
 - 2. Fat/MYVEROL®/Fragrance (60/20/20);
 - 3. PEG 8000/Fragrance (80/20);
 - 4. Fat/SPAN® 40/Fragrance (60/20/20);
 - 5. Fat/SPAN® 40/Fragrance (40/40/20; and
 - 6. Fat/TWEEN® 20/Fragrance (60/20/20).

[0042] The loading of fragrance at 0.3% was run in 1 liter canisters in the launderometer with:

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750 ml water - wash - 30 minutes; and 750 ml water - rinse - 15 minutes.
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[0043] The damp swatches were placed into labeled 568 ml (1 pint) jars where they were rated by panelists.

[0044] Found that most rated No. 4 and No. 5 samples strongest compared to oil on detergent.

45 EXAMPLE II

[0045] Purpose: To determine which carriers perform better in comparison to oil-on-detergent reference in a panel test with expert judges.

Carrier	Description			
0743-1-A	Fat/SPAN® 40/Fragrance (40/40/20)			
0693-1-A	Fat/MYVEROL®/Fragrance (60/20/20)			
0743-1-B	Fat/SPAN® 40/Fragrance (60/20/20)			
0693-1-B	PEG 8000/Fragrance (80/20)			

Fragrance - citrus type laundry fragrance.

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Detergent base - commercial.

Fragrance loading - 0.3%.

Sample preparation:

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- 1. Weighed 0.032 grams of carrier onto 2.23 grams of detergent base, mix thoroughly; and
- 2. Prepared 200 grams of detergent/oil using 0.6 grams fragrance using Turbula mixer for 1/2 hour.

Cloth - 65/35 polyester/cotton swatches.

[0046] Following is a table of panelist responses comparing each carrier to oil-on-detergent reference:

	0743-1-A	0693-1-A	0743-1-B	0693-1-B
Total weaker	7	13	4	13
Much weaker	1	2	0	4
Moderately weaker	5	2	2	3
Slightly weaker	1	9	. 2	6
No difference	0	2	1	2
Total stronger	16	8	11	2
Slightly stronger	9	7	6	1
Moderately stronger	6	1	.4	1
Much stronger	1	. 0	1	0
Percent stronger	70%	38%	73%	13%
Total Panelists	23	23	16	17

[0047] The results show that the two carriers containing SPAN® 40 and fat produced higher sensory intensity results. The Fat/MYVEROL® and PEG 8000 system did not perform well.

COMPARATIVE EXAMPLE III

[0048] Purpose: To evaluate aroma deposition on laundered cloth using carriers.

[0049] <u>Carrier:</u>

0693-1-A - Fat/MYVEROL®/Fragrance (60/20/20); and 0813-1 - SIPERNAT® 22/Fragrance/Fat (40/40.20).

[0050] No. 0813-1 was prepared by absorbing fragrances on SIPERNAT® 22 silica and coating with fat on a fluid bed coater.

Fragrance - citrus type laundry fragrance; and Detergent - with bleach and TAED.

[0051] The detergent base was switched to determine if bleach and TAED influenced carrier performance.

[0052] Table of panelists' responses, comparing each sample to oil-on-detergent reference:

	0693-1-A	0813-1
Total weaker	4	13
Much weaker	1	3
Moderately weaker	1	4

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(continued)

	0693-1-A	0813-1
Slightly weaker	2	6
No difference	0	3
Total stronger	17	5
Slightly stronger	9	3
Moderately stronger	5	1
Much stronger	3	1
Percent stronger	74%	25%
Total Panelists	23	20

[0053] The results show Sample 0693-1-A Fat/MYVEROL® system performed better on the detergent base containing bleach and TAED than the previous run using the non-bleach detergent.

EXAMPLE IV

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[0054] Carriers fabricated containing SIPERNAT® 22 brand silica, fragrance and fat were run previously as carriers. These materials did not perform very well. A carrier containing SIPERNAT® 22 and fragrance coated wit Fat/SPAN® 40 should provide comparison between solid matrix and coated product.

Materials:

[0055] 0833-4

20% SIPERNAT® 22;

40% Fragrance citrus type; and

40% Fat/SPAN® 40.

[0056] This carrier was run on both bleach and non-bleach detergent bases and compared with oil-on-detergent references. This was also compared with solid drum chilled Fat/SPAN® 40/Fragrance which has been shown to be effective

[0057] Cloth (damp) was prepared and submitted to "Sensory Testing Center" (STC) judges for evaluation and the comparison is as follows:

	0833-4/no bleach	0833-4/with bleach
Total weaker	18	2
Much weaker	2	. 0
Moderately weaker	7	. 0
Slightly weaker	9	2
No difference	1	5
Total stronger	2	14
Slightly stronger	2	8
Moderately stronger	0	5
Much stronger	0	1
Percent stronger	10%	88%
Total Panelists	21	21

significantly less effective in the detergent base without the bleach. This effect appears to indicate that the detergent base interaction with the carrier is significant as to effect of the overall deposition profile on cloth. This was also evident in the test with the Fat/MYVEROL® carrier on both bases.

EXAMPLE V

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[0059] Re-evaluated, the Fat/SPAN® carrier is effective on the two detergent bases (with and without bleach): Carrier: 0743-1-A

60% Fat/DURYEY® 17; 20% SPAN® 40; and

20% Citrus Type Laundry Fragrance.

System Label	el Description		
Reference 1	0.3% Fragrance in bleach base		
Sample 1	0743-1-A Carrier in bleach base		
Reference 2	0.3% Fragrance in non-bleach base		
Sample 2	0743-1-A Carrier in non-bleach base		

[0060] The following table lists responses of the panelists, comparing odor intensity of each carrier versus oil-on-detergent on damp cloth in a paired comparison test.

	0743-1 A/bleach	0743-1A/no bleach	0743-1 A/bleach	0743-1 A/no bleach
Total weaker	1	. 8	0 .	2
Much weaker	0	0	- 0	0
Moderately weaker	. 0	1	0	1
Slightly weaker	1	7	. 0	1
No difference	1	4	0	1
Total stronger	20	10	22	19
Slightly stronger	5 .	5	4	7
Moderately stronger	8	4	8	5
Much stronger	7 .	1	10	7
Percent stronger	95%	56%	100%	95%
TOTAL EVALUATIONS	22	22	22	. 22

[0061] The results above show for the damp cloth tested on the bleach base, a consistent positive response by the 22 judges. However, the non-bleach base with the same carrier produced a wider distribution in panelist responses but were still overall positive.

EXAMPLE VI

[0062] The previous test showed positive results for the sample using Carrier 0743-1-A on the bleach base. The non-bleach base has been some concern because of the lack of repeatable results. This test repeats the Carrier 0743-1A on non-bleach base with two sets of panelists. In addition, repetition of the Fat/Fragrance system in Carrier 0693-1-A on the bleach base was done.

[0063] Both carriers contained 20% citrus type laundry fragrance at 0.3% loading.

System Label	Description
Reference 1	0.3% Fragrance on non-bleach base

(continued)

System Label	Description		
Sample 1	Carrier 0743-1-A on non-bleach base		
Reference 2	0.3% Fragrance on bleach base		
Sample 2	Carrier 0693-1-A on bleach base		

	0743-1A/non-bleach	0693-1 A/bleach	0743-1 A/non-bleach	0693-1A/bleach
Total weaker	3	9	5	5 .
Much weaker	1	1	0	0
Moderately weaker	0	5	0	1
Slightly weaker	2	3	5	4
No difference	4	3	2	1
Total stronger	17	12	15	16
Slightly stronger	7	8	7	11
Moderately stronger	· . 7 · .	3	6	3
Much stronger	3	1	2	. 2
Percent stronger	17/20%	12/21%	15/20%	16/21%
TOTAL EVALUATIONS	24	24	22	22

[0064] Compositions were prepared with the following aroma chemicals:

30 LINALYL ACETATE;

ALDEHYDE C;

HEXYL CINNAMIC ALDEHYDE;

CITRAL VA®;

HEXYL SALICYLATE;

35 HELIONAL;

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AMYL SALICYLATE;

LILIAL;

ALLYL AMYL GLYCOLATE;

FLORAL POWDER BQT;

40 ORANGE OIL;

METHYL NONYL ACETALDEHYDE;

PHENYL ETHYL ALCOHOL;

DIHYDRO MYRCENOL;

HEDIONE:

ISO E SUPER®;

TETRA HYDRO LINALOOL

DIHYDRO TERPINEOL; and

GALAXOLIDE®.

[0065] As employed herein and in appended claims, the term "perfume" is used in its ordinary sense to refer to and include any essentially water insoluble fragrant substance or mixture of substances including natural (i.e., obtained by extraction of flowers, herbs, leaves, roots, barks, wood, blossoms or plants), artificial (i.e., a mixture of different nature oils or oil constituents) and synthetic (i.e., synthetically produced) odoriferous substances. Such materials are often accompanied by auxiliary materials, such as fixatives, extenders and stabilizers. These auxiliaries are also included within the meaning of "perfume", as used herein. Typically, perfumes are complex mixtures of a plurality of organic compounds, which may include odoriferous or fragrant essential hydrocarbons, such as terpenes, ethers and other compounds which are of acceptable stabilities in the present compositions. Such materials are either well known in the art or are readily determinable by simple testing, and so need not be listed in detail here.

[0066] The perfumes employed in the invention will preferably be of a polar nature and lipophilic, so that they form at least a significant part of the oil phase of the micro-emulsion. Such perfumes will be hypochlorite-stable, of course, and it has been noted that the best perfumes for this purpose are those which are in the following olfactory families: floral, including floral, green floral, woody floral and fruity floral; chypre, including floral aldehydic chypre, leather chypre and green chypre; fougère; amber, including floral woody amber, floral spicy amber, sweet amber and semi-floral amber; and leather.

[0067] Perfume components and mixtures thereof which can be used for the preparation of such perfumes may be natural products such as essential oils, absolutes, resinoids, resins, etc., and synthetic perfume components such as hydrocarbons, alcohols, aldehydes, ketones, ethers, acids, esters, acetals, ketals, nitriles, etc., including saturated and unsaturated compounds, aliphatic, carbocyclic and heterocyclic compounds. Examples of such perfume components are geraniol, geranyl acetate, linalool, linalyl acetate, tetrahydrolinalool, citronellol, citronellyl acetate, dihydromyrcenol, dihydromyrcenyl acetate, tetrahydromyrcenol, terpineol, terpinyl acetate, nopol, nopyl acetate, 2-phenylethanol, 2-phenylethyl acetate, benzyl alcohol, benzyl acetate, benzyl salicylate, benzyl benzoate, styrallyl acetate, amyl salicylate, dimethylbenzylcarbinol, trichloromethylphenylcarbinyl acetate, p-tert-butyl-cyclohexyl acetate, isononyl acetate, vetiveryl acetate, vetiverol, alpha-n-amylcinammic aldehyde, alpha-hexyl-cinammic aldehyde, 2-methyl-3-(ptert.butylphenyl)-propanal, 2-methyl-3-(p-isopropyl-phenyl)propanal, 3-(p-tert.butylphenyl)propanal, tricyclodecenyl acetate, tricyclodecenyl propionate, 4-(4-hydroxy-4-methylpentyl)-3-cyclohexenecarbaldehyde, 4-(4-methyl-3-pentenyl)-3-cyclohexenecarbaldehyde, 4-acetoxy-3-pentyltetrahydropyran, methyl dihydrojasmonate, 2-n-heptylcyclopentanone, 3-methyl-2-pentyl-cyclopentanone, n-decanal, n-dodecanal, 9-decenol-1, phenoxyethyl isobutyrate, phenylacetaldehyde dimethyl acetal, phenylacetaldehyde diethyl acetal, geranonitrile, citronellonitrile, cedryl acetal, 3-isocamphylcyclohexanol, cedryl methyl ether, isolongifolanone, aubepine nitrile, aubepine, heliotropine, coumarin, eugenol, vanillin, diphenyl oxide, hydroxycitronellal, ionones, methyl ionones, isomethyl ionones, irones, cis-3-hexenol and esters thereof, indane musk fragrances, tetralin musk fragrances, isochroman musk fragrances, macrocyclic ketones, macrolactone musk fragrances, ethylene brassylate, aromatic nitro-musk fragrances. Suitable solvents, diluents or carriers for perfumes as mentioned above are, for example: ethanol, isopropanol, diethylene glycol monoethyl ether, dipropylene glycol, diethyl phthalate, triethyl citrate, etc..

[0068] Among the fatty components that can be used are the following:

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Partially hydrogenated vegetable oils for use in peanut butter (stabilizers), bread production, shortening manufacture, chewing gum base, icings (stabilizer), dry mix, encapsulation, caramel coatings and as a general purpose stabilizing hard fat.

35	Label Ingredient Statement	Stearine 07 Partially Hydrogenated Cottonseed Oil	Stearine 17 Partially Hydrogenated Soybean Oil	Stearine 27 Partially Hydrogenated Palm Oil	K.L.X Partially Hydrogenated Cottonseed, Soybean Oil
	Color	4.0R (Max.)	3.0 OR (Max.)	5.0R (Max.)	2.5R (Max.)
	Free Fatty Acids	0.10% (Max.)	0.10% (Max.)	0.10% (Max.)	0.10% (Max.)
	lodine Value	4.0 (Max.)	4.0 (Max.)	5.0 (Max.)	
40	Capillary				
	Melting Point	(141-147°F)	(152-158°F)	(136-144°F)	(124-130°F)
	Odor/Flavor	Bland	Bland	Bland	Bland
	A.O.M.	-	-	-	200 hours (MIN.)
	Typical Solid Fat	٠.	*.		
45	Index				
	10°C (50°F)		4		80
	21°C (70°F)				80
	27°C (80°F)				80
50	33°C (92°F)	•	,		80
	38°C (100°F)		•	j	.
	43°C (110°F)				62
	Package/Form	Beads in 22,7 kg (50	Flakes in 22,7 kg (50	Beads in 22,7 kg (50	Flakes in 22,7 kg (50
		lb.) cartons, Flakes	lb.) cartons	lb.) cartons, Flakes	lb) cartons
55		in 50 lb. cartons		in 50 lb. cartons	

hydrogenated soybean oil followed by molecular distillation. Typical properties are:

Ту	ype	Fat Source	Monoester Content, Min. %	Propylene Glycol Content, Max. %	Acid Value, Max.	lodine Value, Max.	Specific Gravity at 80°C	Melting Point Approx. °C (°F)	Physical Form
18	806	Hydrogenated Soybean oil	90	1.2	3	5	0.92	69 (156)	Small Beads

[0070] The features disclosed in the foregoing description, in the claims and/or in the accompanying drawings may, both separately and in any combination thereof, be material for realising the invention in diverse forms thereof.

Claims

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- 1. A method for producing an aroma chemical-containing solid particle of improved substantivity for incorporation into laundry detergents characterized in that the process steps, in sequence, consist essentially of:
 - (i) selecting a fat component from the group consisting of glycerol which is mono, di or tri substituted with a saturated or unsaturated organic moiety of 3 to 30 carbon atoms;
 - (ii) heating said fat component to an elevated temperature sufficient to form a first molten melt thereof;
 - (iii) selecting a solid surface active agent from the group consisting of a SPAN® surfactant of HLB 4.3 to 8.6, said SPAN® surfactant being defined as a mixture of compounds having the structure:

wherein R is C₁₁-C₁₇ alkyl or alkenyl;

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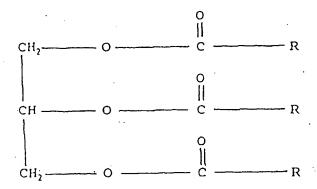
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- (iv) heating said surface active agent to form a second molten melt thereof;
- (v) combining said first and second melts with one or more aroma chemicals and uniformly dispersing said one or more aroma chemicals in the combined melt of said fat component and said surfactant;
- (vi) rapidly cooling said melt to form a solid material containing said fat component, said nonionic SPAN® surfactant and said one or more aroma chemicals; and
- (vii) forming solid particles thereof to obtain an aroma chemical-containing particulate aroma chemical carrier,

wherein the fat component is present in an amount of 40 to 99% by weight, and the surfactant is present in an amount of 1 to 60% by weight, excluding the quantity of aroma chemical, and wherein the aroma chemical is present in an amount of 1 to 40% by weight based on 100 parts by weight of combined fat component and surfactant.

- 2. The method according to Claim 1 further characterized in that the said carrier is added to a laundry detergent base.
- 3. A composition of matter prepared according to the process of Claim 1.
- 4. The composition according to Claim 3 further characterized in that said fat component contains 10 to 20 carbon atoms.
- 5. The composition according to Claim 3 further characterized in that said fat component is at least one natural fat obtained from solid waxy oils obtained, in the alternative, from soybean, palm, corn, cottonseed, safflower and/or coconut plant sources.
 - 6. The composition according to Claim 3 further characterized in that said fat has the formula:



wherein R is the same or different aliphatic organic group containing 5 to 30 carbon atoms.

Patentansprüche

- 1. Verfahren zur Herstellung eines aromachemikalienhaltigen Feststoffpartikels mit verbesserter Substantivität zum Beimengen zu Waschmitteln, dadurch gekennzeichnet, daß die Verfahrensschritte in der Abfolge im wesentlichen daraus bestehen:
 - (i) eine Fettkomponente aus der Gruppe auszuwählen, die aus einfach, zweifach oder dreifach substituiertem Glycerol mit einem gesättigten oder ungesättigten organischen Anteil von 3 bis 30 Kohlenstoffatomen besteht;
 - (ii) diese Fettkomponente auf eine erhöhte Temperatur, die ausreicht, um eine erste geschmolzene Schmelze

davon auszubilden, zu erhitzen;

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(iii) einen festen oberflächenaktiven Stoff aus der Gruppe auszuwählen, die aus einem SPAN® oberflächenaktiven Stoff mit 4,3 bis 8,6 HLB besteht, wobei dieser SPAN® oberflächenaktive Stoff als ein Gemisch von Verbindungen definiert ist, die die Struktur haben:

worin R C₁₁-C₁₇ Alkyl oderAlkenyl ist;

- (iv) diesen oberflächenaktiven Stoff zu erhitzen, um eine zweite geschmolzene Schmelze davon auszubilden;
- (v) die ersten und zweiten Schmelzen mit einer oder mehreren Aromachemikalie/n zu verbinden und die eine oder mehreren Aromachemikalie/n gleichmäßig in der Verbundschmelze von Fettkomponente und oberflächenaktivem Stoff fein zu verteilen;
- (vi) die Schmelze zur Ausbildung einer festen Substanz, die die Fettkomponente, den nichtionischen SPAN® oberflächenaktiven Stoff und die eine oder mehrere Aromachemikalie/n enthält, schnell abzukühlen; und
- (vii) Feststoffpartikel davon auszubilden, um eine aromachemikalienhaltige, aus einzelnen Teilchen bestehende, aromachemische Trägersubstanz zu gewinnen;

in der die Fettkomponente in einer Menge von 40 bis 99 Gew.-% vorhanden ist; der oberflächenaktive Stoff, ohne die Menge an Aromachemikalie, in einer Menge von 1 bis 60 Gew.-% vorhanden ist, und in der die Aromachemikalie in einer Menge von 1 bis 40 Gew.-%, basierend auf 100 Gewichtsteilen der Verbindung aus Fettkomponente und oberflächenaktivem Stoff, vorhanden ist.

2. Verfahren nach Anspruch 1, ferner dadurch gekennzeichnet, daß die Trägersubstanz einem Waschmittelgrundstoff beigefügt wird.

- 3. Eine nach dem Verfahren von Anspruch 1 hergestellte Stoffverbindung.
- 4. Zusammensetzung nach Anspruch 3, ferner dadurch gekennzeichnet, daß die Fettkomponente 10 bis 20 Kohlenstoffatome enthält.
- 5. Zusammensetzung nach Anspruch 3, ferner dadurch gekennzeichnet, daß in der Fettkomponente mindestens ein natürliches Fett enthalten ist, das aus festen Wachsölen, wahlweise aus Sojabohnen, Palmen, Weizen, Baumwollsamen, Färberdistel und/oder Kokosnuß pflanzlicher Herkunft gewonnen wurde.
- 10 6. Zusammensetzung nach Anspruch 3, ferner dadurch gekennzeichnet, daß das Fett die Formel hat:

in der R dieselbe oder eine unterschiedliche alipathisch organische Gruppe ist, die 5 bis 30 Kohlenstoffatome enthält.

30 Revendications

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- Procédé de production d'une particule solide contenant un arôme chimique de tenue améliorée à incorporer dans des lessives caractérisé en ce que les étapes du procédé, successivement, consistent sensiblement à :
 - (i) choisir un composant gras dans le groupe constitué du glycérol qui est mono, di ou tri-substitué avec un fragment organique saturé ou insaturé de 3 à 30 atomes de carbone;
 - (ii) chauffer ledit composant gras à une température suffisamment élevée pour former un premier mélange fondu de celui-ci;
 - (iii) choisir un agent tensioactif solide dans le groupe constitué d'un tensioactif SPAN® de HLB 4,3 à 8,6, ledit tensioactif SPAN® étant défini comme un mélange de composés ayant la structure :

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dans laquelle R est un groupe alkyle ou alcényle en C_{11} à C_{17}

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- (iv) chauffer ledit agent tensioactif pour former un second mélange fondu de celui-ci;
- (v) mélanger lesdits premier et second mélanges fondus avec un ou plusieurs arômes chimiques et disperser uniformément lesdits un ou plusieurs arômes chimiques dans le mélange fondu réuni dudit composant gras et dudit tensioactif;
- (vi) refroidir rapidement ledit mélange fondu pour former un matériau solide contenant ledit composant gras, ledit tensioactif SPAN® non ionique et lesdits un ou plusieurs arômes chimiques; et
- (vii) former des particules solides de celui-ci pour obtenir un vecteur particulaire contenant un arôme chimique,

dans lequel le composant gras est présent en une quantité de 40 à 99 % en poids, et le tensioactif est présent en une quantité de 1 à 60 % en poids, à l'exclusion de la quantité d'arôme chimique, et dans lequel l'arôme chimique est présent en une quantité de 1 à 40 % en poids pour 100 parties en poids de composant gras et de tensioactif réunis.

- 2. Procédé selon la revendication 1, caractérisé en outre en ce que ledit vecteur est ajouté à une base détergente de lessive.
- 3. Composition de matière préparée selon le procédé de la revendication 1.
 - 4. Composition selon la revendication 3 caractérisée en outre en ce que ledit composant gras contient 10 à 20 atomes de carbone.
- 5. Composition selon la revendication 3, caractérisée en outre en ce que ledit composant gras est au moins une graisse naturelle obtenue à partir d'huiles cireuses solides obtenues, au choix, à partir de sources végétales de soja, palme, maïs, graines de coton, carthame et/ou noix de coco.
 - 6. Composition selon la revendication 3 caractérisée en outre en ce que ladite graisse a la formule :

dans laquelle R peut être un groupe organique aliphatique identique ou différent contenant 5 à 30 atomes de carbone.